



Site-Wide Sampling and Analysis Plan Delaware City Refinery RCRA Facility Investigation, Post-Closure and Corrective Action Program Revision 1

Prepared for:
Motiva Enterprises Delaware City, Delaware

Submitted to:
Delaware Department of Natural Resources and Environmental Control
Dover, Delaware

Prepared by:
Golder Associates Inc.
11231 Richmond Ave, Suite D104,
Houston, Texas, USA 77082
+1 832 916-3690

March 2019

Signature Page

Christopher Brown, P.G.
Delaware Department of Natural Resources and Environmental Control
Case Manager

Alison Kiliszek, EIT.
Delaware Department of Natural Resources and Environmental Control
Case Manager

Robert Jennings, P.E.
Motiva Enterprises, LLC
Project Coordinator

George Arbutina
Remedial Construction Services, L.P.
Project Director

Sean Reilly
Remedial Construction Services, L.P.
Project Manager

Brenda Basile
Golder Associates Inc.
Quality Assurance Officer

Table of Contents

1.0 INTRODUCTION	1
1.1 Objectives.....	1
1.2 Project Organization.....	1
2.0 SITE BACKGROUND	2
2.1 Previous Investigations	4
2.2 Geologic and Hydrogeologic Information	4
3.0 SAMPLING RATIONALE	4
4.0 FIELD METHODS AND PROCEDURES.....	5
4.1 Field Equipment and Supplies	5
4.2 Calibration of Field Equipment	6
4.2.1 Multi-Parameter Water Meter.....	6
4.2.2 Photoionization Detector (PID).....	6
4.2.3 Calibration Records	6
4.3 Field Screening	6
4.4 Sample Procedures and Equipment	7
4.4.1 Soil	7
4.4.2 Soil Gas.....	11
4.4.3 Leachate Sampling	12
4.4.4 Sediment Sampling.....	14
4.4.5 Surface Water and Stormwater.....	15
4.4.6 Groundwater	15
4.5 Field Quality Control Samples.....	22
4.5.1 Assessment of Field Contamination	22
4.5.2 Assessment of Field Variability (Field Duplicates)	22
4.5.3 Assessment of Matrix Effects.....	23
4.6 Decontamination	23

4.6.1	Surface/Sediment/Subsurface Soil Sampling Equipment.....	23
4.6.2	Groundwater Equipment.....	24
5.0	SAMPLE CONTAINERS, PRESERVATION AND ANALYSIS	24
6.0	SAMPLE DOCUMENTATION AND SHIPPING	25
6.1	Field Log Books/Field Sampling Forms	25
6.2	Labeling.....	26
6.3	Sample Chain-of-Custody Forms and Custody Seals	26
6.4	Packaging and Shipment.....	27
7.0	IDW DISPOSAL	27
8.0	FIELD VARIANCES	28
9.0	FIELD HEALTH AND SAFETY PROCEDURES.....	28
10.0	REFERENCES	28

TABLES

Table 1: Analyses, Methods, Sample Containers, and Preservation/Holding Time Requirements

FIGURES

Figure 1: Example Field Data Sheet

Figure 2: Example Sample Label

Figure 3: Example Chain-of-Custodies

Figure 4: Example Custody Seal

1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) establishes procedures for investigation and corrective-action activities conducted at the Delaware City Refinery (Refinery) in Delaware City, Delaware. Sampling activities are conducted to support the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) as well as groundwater compliance and corrective-action monitoring. This SAP addresses tasks related to RCRA Corrective Action and Closure of Solid Waste Permitted Units at the Refinery. Sampling activities are conducted in response to the requirements of permits, corrective-action programs, and agreed orders from the Delaware Department of Natural Resources and Environmental Control (DNREC).

Associated quality assurance requirements are described in the Quality Assurance Project Plan (QAPjP) and data-management procedures are described in the Data Management Plan. The project Health and Safety Plan (HASP) is an integral component of the SAP. The plans will be reviewed periodically for effectiveness and efficiency and will be modified as appropriate. Updates to the SAP will be submitted to DNREC.

Details for sample locations, specific sampling protocols, analytes, and other requirements will be specified in the task-specific work plans such as the Groundwater Monitoring Plan or RFI Work Plans. This SAP will be an appendix to the task-specific work plans.

1.1 Objectives

The objectives of this SAP are to:

- Describe the project background;
- Present the project data-quality objectives;
- Describe the sampling rationale;
- Describe sampling methods and documentation;
- List quality-control requirements; and
- Describe corrective-action procedures.

1.2 Project Organization

The agency responsible for the project is DNREC. All work related to this project must be approved either by DNREC or by the Refinery. Work-related items not performed under the direction of DNREC are to be performed under the direction of United States Environmental Protection Agency (USEPA) Region III.

Title/Responsibility	Name	Affiliation	Phone Number
DNREC Case Manager (Hazardous Waste)	Christopher Brown, P.G.	DNREC	302-739-9403
DNREC Case Manager (Solid Waste)	Alison Kiliszek, EIT	DNREC	302-739-9403
EPA Case Manager	Barbara Smith	USEPA Region III	215-814-5786
Project Coordinator	Robert Jennings	Motiva Enterprises, LLC	713-427-3574

Title/Responsibility	Name	Affiliation	Phone Number
Delaware City Refinery Coordinator	Thomas Godlewski	PBF Energy	302-757-1192
Project Director	George Arbutina	Remedial Construction Services, LP	484-367-5422
Project Manager	Sean R. Reilly	Remedial Construction Services, LP	484-367-5423
Quality Assurance Officer	Brenda Basile	Golder Associates Inc.	832-916-3691
Site Superintendent	John Hunsberger	Remedial Construction Services, LP	484-367-5400
Health and Safety Officer	John Hunsberger	Remedial Construction Services, LP	484-367-5400

2.0 SITE BACKGROUND

The Refinery is located in Delaware City, Delaware. The site is bordered by industrial complexes and farmland to the north, farmland to the south, the Delaware River to the east, and industrial complexes and farms to the west.

The Refinery was originally owned and operated by Tidewater Oil Company. The refining operations occupy about 1,000 acres. The facility processes a variety of crude oils and currently produces about 180,000 barrels of petroleum product a day. Production has included diesel, gasoline, jet, fuel oil, aromatics, methanol, and propane fuels. A timeline of the Refinery operations includes:

1957: Tidewater Oil opens Delaware City Refinery to process high-sulfur crude oil.

1967: Tidewater Oil and Getty Oil merge; Delaware City Refinery takes on Getty name.

1984: Texaco purchases Getty Delaware site.

1988: Texaco sells a share of its Delaware refining operation to Saudi Arabia's national oil company, renaming the plant Star Enterprise.

1998: Shell Oil purchases a 35 percent share of Star Enterprise. Delaware City Refinery and other group holdings become part of Motiva Enterprises.

2004: Independent refiner Premcor Inc. purchases Delaware City Refinery as the company's fourth wholly owned operation.

2005: Valero Energy purchases Premcor's four refineries, including the Delaware City Refinery.

2010: Valero Energy sells the Delaware City Refinery to PBF Energy Partners LP (PBF). PBF continues to operate the Delaware City Refinery as of the date of this SAP.

DNREC administers the RCRA Corrective Action program for the site under Corrective Action Permit HW-09A13, in accordance with Title 7 of the Delaware Code, and the Delaware Regulations Governing Hazardous Waste, with support provided by the USEPA. Post-closure activities are addressed under Hazardous-Waste permits 16-HW-12 (Land Treatment Unit [LTU]), HW-09A14 (Hazardous-Waste Landfill [HWL]), and HW-08A06 (Corrugated

Plate Interceptor [CPI] Surge Basin, and Solid-Waste permits SW-04/01 (Industrial-Waste Landfill [IWL]) and SW-96/01 (Fly-Ash Pond [FAP]).

As the data indicate a need or as required by permit, corrective-action program, or agreed order, the following activities may be performed at the Refinery for the RFI Investigation and corrective-action program:

- Investigation of solid waste management units
- Implementation of the Phase II RFI Work Plan
- Groundwater plume delineation
- Surface water and sediment sampling
- Groundwater monitoring
- Groundwater remediation
- Groundwater flow modeling
- Hydrocarbon recovery (measurements and evacuation of wells and storage tanks from skimmers which have free phase hydrocarbon)
- Stormwater sampling
- Leachate sampling
- Soil sampling
- Soil gas sampling
- Surveying
- Well maintenance and closures
- Waste management and coordination for disposal
- Database management
- Risk assessment
- Daily monitoring of wildlife
- Addressing chlorinated hydrocarbon issues from the Refinery or adjacent site(s)
- Permitting activities
- Associated sampling programs and stormwater issues associated with closures
- Routine monitoring and reporting of the closed Hazardous Waste Landfill, Land Treatment Unit, and CPI Surge Basin
- Developing various protocols and strategies

- Interaction with agencies, management of subcontractors, including drillers, surveyors, laboratories, construction firms, waste transportation and disposal contractors.

2.1 Previous Investigations

Detailed descriptions of previous work (i.e., investigations, studies) performed on site have been provided in the associated task-specific reports, which have been submitted to DNREC and USEPA. Descriptions of current and planned work have been presented in the Phase II RFI Work Plan and will be presented in task-specific work plans as they are developed.

2.2 Geologic and Hydrogeologic Information

The Refinery lies within the Atlantic Coastal Plain physiographic province, approximately 12 miles south of the Piedmont physiographic province boundary, also known as the Fall Line. The Coastal Plain in this region consists of a wedge of unconsolidated sediments deposited on a seaward (southeasterly) sloping crystalline basement. The sediments underlying the DCR have been divided into eight geologic units ranging in age from recent (Quaternary) to more than 65 million years old (Cretaceous). The four most prevalent and pertinent units at the Refinery are the Columbia, Merchantville, and Potomac Formations, and the Riverine deposits. The other four (Marshalltown, Mount Laurel, Englishtown, and Magothy) are locally rare, limited in vertical and lateral extent, and have very little effect on the hydrogeology of the Refinery.

The two major aquifers in the Refinery area are the Columbia and Potomac Formations. The Merchantville Formation, which separates the Columbia and Potomac Formations, generally acts as a confining unit beneath most of the Refinery due to its fine-grained composition. The saturated thickness of the Columbia Aquifer varies from 10 to 100 feet beneath the refinery.

Surface water bodies in the vicinity of the Refinery consist of the following.

1. Delaware River which borders the eastern edge of the Refinery and exhibits tidal characteristics. The Delaware River is not used a source of drinking water but is used as an industrial water source.
2. Dragon Run Creek south of the Refinery and its unnamed tributaries. These are tributaries of the Delaware River.
3. Red Lion Creek north of the Refinery and its unnamed tributaries. These are tributaries of the Delaware River.

Surface water runoff is, for the most part, diverted to the stormwater sewer system, which carries the runoff to a number of permitted stormwater outfalls or to the wastewater treatment plant.

The regional and Refinery-specific geology and hydrogeology are discussed in detail in the Phase II RCRA Facility Investigation Groundwater (URS, 2011). Geology and hydrogeology specific to waste units are discussed in the permits and post-closure plans.

3.0 SAMPLING RATIONALE

The rationale for sampling events is typically determined by the respective permits issued by DNREC, by on-going site investigations, or by previous site investigations, with sampling rationales presented in each task-specific work plan. In-depth explanations of sampling rationales are presented in each task-specific work plan.

4.0 FIELD METHODS AND PROCEDURES

This section describes the methods and procedures for sampling soil, sediment, groundwater, surface water, leachate, soil gas, and investigation-derived waste (IDW).

4.1 Field Equipment and Supplies

Field equipment and supplies are stored on the on-site trailer when not in use during sample events. Below is a list of generic sampling equipment. Media-specific equipment is listed in the sample equipment and procedures section (Section 4.4).

- Decontamination equipment and fluids;
- Plastic sheeting;
- Ice;
- Sample bottles, labels, packing material, custody seals, cooler shipping labels, coolers, and chain-of-custody (C-O-C) forms;
- Stakes, marking flags, and/or paint;
- Field log book and field sample forms;
- Required health and safety clothing and equipment (personal protective equipment [PPE]);
- Packing tape;
- Paper towels or disposable rags;
- Permanent marking pens;
- Watch/clock;
- Safety knife;
- Trash bags;
- Ruler or tape measure;
- Photoionization detector (PID) with 10.6 electron-volt (eV) lamp and 100 parts per million (ppm) isobutylene calibration gas
- Cones, caution tape, and traffic vests;
- Fire extinguisher and first aid kit;
- Investigation-derived waste containers and labels;
- Sampling and Analysis Plan;
- Names and phone numbers of important project and subcontractor contacts;
- Maps of sampling locations and significant site features (such as the locations of utilities);

- Applicable permits; and
- Health and Safety Plan, and documentation including emergency numbers, maps and directions to nearest hospital/health center, and Safety Data Sheets for chemicals of concern.

4.2 Calibration of Field Equipment

Field instrumentation shall be calibrated or verified under controlled conditions prior to field use utilizing manufacturer's recommended operating procedures. Daily performance checks shall be conducted prior to the start of each sampling day and at the end of each sampling day. Field parameters must be within $\pm 10\%$ of the known concentration prior to begin sampling. Instrument calibration must be recorded in a field-sampling logbook or on a field instrument calibration sheet. A reserve, or backup, instrument shall be kept with the sampling team in case any problems arise with the first instrument. The reserve instrument shall be calibrated the same way, and only needs to be checked if the backup instrument is to be placed in service. The field sampling crew shall make note of any instrument change in the field sampling book.

4.2.1 Multi-Parameter Water Meter

A multi-parameter water meter, such as those manufactured by YSI or Horiba, is a multi-functional instrument capable of measuring pH, conductivity, dissolved oxygen, oxidation-reduction potential (ORP), turbidity, and temperature. Each parameter has its own set of calibration standards for initial calibration and for re-calibration of the instrument. The instrument is calibrated at the beginning of each work day, and the calibration is checked at the end of the work day. Should a field calibration check fall outside the normal acceptance criteria, the instrument must be re-calibrated before taking any more measurements. If the instrument cannot be re-calibrated, the meter will be returned to the manufacturer for service and/or recalibration. pH is reported in standard pH units; conductivity is reported in microsiemens (μS); dissolved oxygen is reported in milligrams per liter (mg/L); ORP is reported in millivolts (mV); turbidity is reported in nephelometric turbidity units (NTUs); and temperature is reported in degrees Celsius ($^{\circ}\text{C}$).

4.2.2 Photoionization Detector (PID)

Prior to daily use, the PID is spanned against a zero gas and a 100-ppm isobutylene gas. Should a field check fall outside normal acceptance criteria, the meter will be returned to the manufacturer for service and/or recalibration. Field conditions, such as changing humidity and temperature, may require the PID to be recalibrated. PID readings will be reported in ppm.

4.2.3 Calibration Records

Each sampling team will maintain a field log book or field sampling sheets for calibration records. At the end of each phase of the investigation or groundwater monitoring event, the field sampling team will deliver copies of field log book pages, field sampling forms, and sample collection forms completed during that phase of the investigation to the Field Investigation Manager. Documents and recordkeeping are discussed in the Site-Wide Quality Assurance Project Plan for the Delaware City Refinery RFI, Post-Closure and Corrective Action Program.

4.3 Field Screening

Soil borings will be continuously logged for lithology and soil cores from each borehole will be examined and described by a qualified field geologist. Unless otherwise specified in the task-specific workplan, the Unified Soil Classification System (USCS) will be employed. In addition, soil core samples will be visually inspected for NAPL presence or other potential indications of contamination.

Field samples will be checked for the presence of organic vapors using a PID. Soil headspace samples will be collected as described in the task-specific work plan and screened in the field for total organic vapor concentrations using the PID. These PID readings will be recorded in the field log book or field sampling forms. If specified in the task-specific work plan, PID readings can aid in selecting samples for laboratory analysis. The headspace sample will not be submitted to the laboratory for analysis. The headspace of groundwater monitoring wells will be measured using a PID immediately after removing the well casing cap.

The background concentration of organics in air (the readings for ambient air in the breathing zone, which in most cases should be at or near zero) as measured with the PID will be determined each day and recorded in the field log book prior to the commencement of field activities. The air in the breathing zone will be monitored with the PID throughout intrusive activities. Action levels for upgrading PPE will be specified in the HASP.

4.4 Sample Procedures and Equipment

This section describes procedures for sampling surface and subsurface soil, sediment, groundwater, surface water, leachate, soil gas, and IDW.

4.4.1 Soil

Recommended soil-sampling locations will be identified in the task-specific work plans. If needed, field personnel adjust sampling locations with the approval of the Project Manager. Soil-sample location data will be recorded in the field log book as sampling is conducted. In addition to the generic equipment listed in Section 4.1, sampling equipment for soil media are listed below:

- Bucket augers;
- Hollow-stem auger drill rig, direct-push drill rig, rotosonic drill rig, mud rotary, and tripod drill rig; as well as all associated drilling equipment and tools (subcontractor);
- Three-gas meter with lower explosive limit (LEL), hydrogen sulfide (H₂S), and carbon monoxide (CO) detectors (subsurface work inside Refinery only);
- Hand trowels (either disposable or reusable);
- Stainless-steel or disposable aluminum mixing/homogenization bowls;
- Soil-boring logs;
- T-handle for Encore™ samples or scale and subcoring device for methanol preservation of volatile organic compound (VOCs) (alternative VOC sampling devices such as Terracore™ kits are also acceptable); and
- Munsell Color Plates

Surface Soil

Surface-soil samples shall be collected from the upper 6 inches, or as indicated by the DNREC-approved task-specific work plan. Surface-soil samples for VOC analyses will be collected using a 5-gram Encore™ sampling device or Terracore™ kits. Encore samples will be placed in their original pouches and then sealed in plastic bags before being placed into an ice-filled cooler. Encore samples from different sample locations will not be placed into the same plastic bag. Terracore vials will be placed into the original holder and then sealed in plastic bags before being placed into an ice-filled cooler. Terracore samples from different sample locations will not be

placed into the same plastic bag. The field identification, date and time sampled, and sampler initials must be entered on the vial label, which has the tared vial weight. Do not place a second label on the vial.

Sample containers for the remaining analyses should be completely filled following homogenization of the soil. If sample material is limited, the Project Manager and the laboratory should be contacted to determine the minimum quantity required for analyses. Lid threads should be cleaned prior to closing the sample container to prevent potential contaminant migration to or from the sample, and to prevent container leakage during shipment. Sample containers will be closed as soon as they are filled, chilled to $\leq 6^{\circ}\text{C}$ if appropriate, and processed for shipment to the laboratory.

Bucket-Auger Techniques

Bucket-auger borings will be advanced with a decontaminated high carbon or stainless-steel auger bucket, rods, and t-handle. Decontamination of the auger bucket will be performed prior to sample collection after reaching the target sampling depth. If unsatisfactory sample recovery occurs at the desired sampling interval, the boring location will be offset a maximum of 3 feet and re-sampled at the target interval. The soil aliquots for analyses will be collected as specified in the task-specific work plan. As described above, soil for VOC analyses will be placed directly into the appropriate sample containers. The use of solid-wall bucket augers for collecting soil for VOC analyses will be evaluated on a case by case basis and will consider conditions such as, but not limited to, lithology of soils to be sampled, site access, and depth of sampling. Soil needed for other analyses will be homogenized and placed directly into appropriate laboratory sample containers.

Subsurface Soil

The use of hollow-stem auger, mud-rotary, rotosonic, and/or direct-push drilling techniques is anticipated for the majority of borehole drilling. Each boring will be drilled to the depth specified for sample collection. In general, soil samples will be collected using the following protocols once brought to the surface: The samples collected from each boring will be evaluated for evidence of environmental impact through visual observations, olfactory evidence, and PID readings. For each boring, one discrete sample will be collected from each depth interval exhibiting evidence of contamination, or as otherwise specified in the task-specific work plan. If no contamination is evident, soil samples will be collected from a depth as specified in the task-specific work plan.

Samples for VOC analyses will be collected immediately as described for surface soil. Samples for other analyses will be placed into a dedicated or decontaminated pan, homogenized, and then placed into appropriate laboratory containers. Lid threads should be cleaned prior to closing the sample container to prevent potential contaminant migration to or from the sample and to prevent container leakage during shipment. Sample containers will be closed as soon as they are filled, chilled to $\leq 6^{\circ}\text{C}$, if appropriate, and processed for shipment to the laboratory.

Direct-Push Techniques

Direct-push drilling techniques (e.g., Geoprobe®) use a hydraulic system to push and/or hammer a sampling device into the ground without the use of casing or augers. Two-inch outside diameter macro-core sampler barrels with acetate sample liners installed inside them will be used for sample collection. If unsatisfactory sample recovery occurs at the desired sampling interval, the boring location will be offset no more than 5 feet, a maximum of 3 times, and re-sampled at the desired interval. After a maximum of three attempts, the field sampling team shall contact the Project Manager and discuss potential alternative methods and techniques for soil collection.

based on observed subsurface conditions. Alternative methods and techniques must be approved by the DNREC Case Manager prior to implementation.

The procedures for obtaining soil samples include the sampler being removed from the borehole, the acetate liner containing the soil sample core being extruded from the macro-core sampler, and the acetate liner being opened with a cutting tool. Acetate sample liners will be opened length-wise by the drilling subcontractor (using a safety [shielded or retractable blade] knife or equivalent suitable equipment) for inspection and sampling. The acetate liner containing the soil samples will be placed on a clean surface for geologic logging and sampling.

To protect sample integrity, each of the samplers will be fitted with a new macro-core acetate liner prior to each use. Any non-dedicated soil sampling equipment and down-hole drilling equipment will be decontaminated prior to each use.

Hollow-Stem Auger

Hollow-stem auger drilling is performed using a rotary drilling rig and hollow-stem augers to drill to the “target” depth in unconsolidated soils. The inside diameter of the augers will be determined based on the goals of the drilling program (e.g., soil sampling only or installation of a 2-inch or 4-inch well). A center bit or auger plug should be used to prevent soils from entering the inside of the auger. During hollow-stem auger drilling, split-barrel samplers are typically used to collect soil samples from undisturbed soils beneath/ahead of the lead auger. The frequency of split-barrel sampling should be specified in the task-specific work plan and will typically be dependent on project goals (e.g., soil contaminant evaluation, new well installation, damaged well replacement, etc.), current understanding of soil lithology and boring density within an area of interest. If excessive amounts of soil enter the center of the augers prior to split-barrel sampling, this material should be removed from the augers to allow collection of representative soil samples.

Mud Rotary Drilling

Mud-rotary drilling is used when borehole walls are very unstable, conditions frequently encountered in non-cohesive sand units. Tri-cone roller bits are used to perform the drilling. A dense drilling fluid (a mixture of water and bentonite, Revert®, or other synthetic drilling products) is used to stabilize the boring walls and to remove the drill cuttings from the boring. Types of drilling-fluid additives should be specified in the work plan and approved by the Project Manager. A strainer is held in the drill-water discharge stream to catch the suspended material (drill cuttings). The weight of the fluid and the quantity of water pumped through the bit will determine the size of the particles that can be removed from the boring with the circulating fluid. Split-barrel samplers are used for collecting soil samples. The amount of sample necessary at each depth interval will dictate the dimensions of the split-barrel sampler (2-inch by 2-foot or 3-inch by 1.5-feet). Soil-sample intervals for laboratory analyses will be selected based on the criteria outlined in each task-specific work plan.

Rotosonic Drilling

The rotosonic drilling method allows for continuous soil sampling with high sample integrity by first advancing an inner 4-inch ID by 10-foot-long core barrel. Once the core barrel has reached the bottom of the next 10-foot soil sampling interval, an outer steel-casing is advanced to the same depth as the lead end of the inner core barrel. The diameter of the outer casing to be used is dependent upon subsurface conditions such as the presence of confining units that will be penetrated, the use of the boring for soil sampling, or the diameter of monitoring wells. Soil in front of the outer-casing drill bit is removed or displaced using a combination of water, injected under high pressure through orifices within the drill bit, and the rotational and vibrational forces of the drill bit and casing. To

obtain the soil samples, the core barrel is removed from within the outer casing, and the sample material is vibrated out of the core barrel, into 6-inch diameter by 10-foot plastic bags.

Boring Abandonment

At the conclusion of each boring, the borehole will be filled with either the soil cuttings, bentonite hole plug, or with a cement/bentonite grout using the tremie-method. The specific method of borehole backfilling will be dependent on the borehole depth, presence of groundwater, contaminants encountered, etc., as presented in the task-specific work plan. If a grout is used, the augers/casing and borehole are completely filled with the grout from the bottom up with a tremie-pipe. Grout will be of sufficient density to lift water without excessive mixing. During grouting, the top of the grout column is maintained near ground level as the augers/casings are withdrawn from the borehole and removed from the drill string. The borings will be topped off with additional grout or concrete, to grade, as late in the drilling program as possible if grout settlement has occurred.

Test Pit and Post-Excavation Sampling

This section describes procedures for collection of samples from test pits or excavations. Because of accessibility and safety concerns, and for ease of sampling, a hydraulic excavator equipped with a power shovel will be used to reach sample locations within test pits and excavations. Once the location and target depth for soil sampling is selected, a backhoe shovel will be used to obtain soil from that location within the test pit or excavation.

Sample(s) will be collected from the approximate center of the soil in the backhoe shovel to ensure that the sample has not come in contact with or been contaminated by the shovel. Samples for VOC analyses will be collected immediately, as described for surface soil. Samples for other analyses will be placed in a dedicated or decontaminated pan, homogenized, and then placed into appropriate laboratory containers. Sample containers will be closed as soon as they are filled, chilled to $\leq 6^{\circ}\text{C}$, if appropriate, and processed for shipment to the laboratory.

Following collection of each sample, any portion of the shovel load not selected for laboratory analysis will be placed onto a soil stockpile. No stockpiled soils will be placed directly on soil or vegetative surfaces; soil stockpiles will be placed on plastic sheeting. If it is determined that excavated and stockpiled soil from the test pit soil has acceptably low levels of constituents, test pits will be backfilled with this stockpiled soil. All impacted soils not appropriate for use as backfill will be placed in appropriate containers, such as lined roll-off boxes for off-site disposal. All stockpiled and containerized waste soils will be covered at the end of each day. Soils for disposal will be profiled for waste characteristics as described below.

Soil Waste-Characterization Sampling

Waste materials such as drill cuttings and impacted trash or protective equipment will be temporarily stored on-site and containerized in either 55-gallon DOT approved drums or lined roll-off boxes. The areas for temporary storage will be the onsite 90-day storage pad at the wastewater treatment plant. Waste containers can also be placed near the work area for short periods while work is being performed but must be removed to the temporary storage areas once work is completed. The drum identification, which will be kept on a log in the site office trailer, will consist of a unique 6-digit number which corresponds to the date of generation (i.e., September 3, 2019, would be labeled 090319) followed by container sequence (01, 02, 03, etc.). An example of a complete identification is 090319-01. A copy of this log will be sent to the Project Manager each week during field activities.

If soil cuttings are disposed of in roll-off container(s), a composite of the contents of each roll-off will be collected. If material is in drums, a representative composite of the cuttings will be collected for each specific investigation area.

Solid wastes generated as a result of work performed at the refinery have generally been pre-classified as non-hazardous. Containerized solids will be transported off-site following sampling, characterization, approval of the waste disposal facility, and proper manifesting. The soil will be manifested (signed by Motiva or designee as the generator) and shipped via a DOT approved waste carrier to an approved treatment, storage and disposal facility.

Samples for VOC analyses will be collected using a 2-ounce jar, completely filled with no headspace remaining between the sample surface and the jar lid. Sample containers will be closed as soon as they are filled and chilled immediately to $\leq 6^{\circ}\text{C}$. Sample containers for the remaining analyses should be completely filled following homogenization of the soil. Lid threads should be cleaned prior to closing the sample container to prevent potential contaminant migration to or from the sample and to prevent container leakage during shipment. Sample containers will be closed as soon as they are filled, chilled to $\leq 6^{\circ}\text{C}$, if appropriate, and processed for shipment to the laboratory.

4.4.2 Soil Gas

In addition to the generic equipment listed in Section 4.1, the sampling equipment for soil gas and ambient air samples is listed below.

- 6-liter Summa™ canisters equipped with flow regulators;
- Helium tank and helium meter;
- Shroud for QA/QC testing prior to sampling;
- Dedicated Teflon™-lined polyethylene tubing;
- Peristaltic pump or vacuum pump; and
- Landfill Gas or oxygen (O_2)/carbon dioxide (CO_2) Meter

The DNREC Site Investigation and Restoration Section standard operating procedure “Active Soil Gas Sampling Procedure” provides details for installing and sampling active soil gas monitoring points. The task-specific work plan will specify these procedures or propose alternative methods for approval by DNREC.

Soil gas samples are collected using 6-liter Summa™ canisters. The canisters will be cleaned and certified by the laboratory prior to use. The canister is evacuated to 30 inches Hg by the laboratory (i.e., negative 30 inches Hg). A flow control valve is attached to the canister, which regulates the amount of air sampled per hour up to a certain total number of hours (time-weighted average). A Teflon™-lined tube is attached to the end of the regulator and positioned at the sampling point. If air sampling is to be performed in an open area (i.e., not a boring hole), the Teflon™-lined tube is generally placed and supported approximately four to five feet above ground level. If necessary, the sampling equipment should be set up to prevent moisture or other foreign debris from entering the sample collection tubing. If soil gas sampling is performed in a boring hole, the sampling point is sealed to prevent ambient air from entering the boring hole and three to five volumes of air is then purged with a peristaltic pump before sampling begins. One volume of air is the volume of the inside of the boring hole and the volume of the tubing. Sampling points are monitored using a landfill gas meter or O_2/CO_2 meter during sampling.

The canister should be checked every twenty minutes initially to ensure that the pressure inside the canister continues to increase at a constant rate. Once the canister pressure has reached a pressure of negative 5 inches Hg, the flow valve can be shut off and the Summa™ canister is processed for shipment to the laboratory.

Weather conditions and field parameters (such as O₂/CO₂) during sample collection are recorded on the field sampling forms. The initial and final times and pressure readings are recorded on the C-O-C and field sampling forms. Flow control valves and pressure regulators are removed. Valves, pressure regulators, and Summa™ canisters are placed in the shipping box for return to the analytical laboratory. Samples are shipped under C-O-C procedures.

4.4.3 Leachate Sampling

Samples are collected from the IWL and FAP leachate collection system (LCS) and leak detection system (LDS) as specified in the post-closure care plans. In addition to the generic equipment listed in Section 4.1, sampling equipment for leachate media are listed below.

- Bladder pump and control box;
- Battery or gasoline powered generator for source of electric power;
- Dedicated Teflon™-lined bladders;
- Containers for containerizing water; and
- Paper towels, trash bags, small plastic bags, packing tape;

Water level measurements are not performed. The leachate collection system is not purged and field parameters are not recorded prior to collecting leachate samples. Grab samples are collected for analyses specified in the post-closure care plans. Leachate samples for the LDS and LCS are collected from sample ports using dedicated submersible pumps for each system. FAP leachate samples are collected from the LDS sumps via sample ports located at the top of each sump using dedicated submersible pumps.

Sampling, sample handling, and decontamination will be conducted with appropriate PPE based on the constituents known to exist in the groundwater (based on historical groundwater data or refinery process data, where available). Purge water and decontamination water will be containerized and transported to the Refinery WWTP for disposal upon approval by the WWTP Operator. Water will not be discharged onto the ground surface unless prior approval is given by the Refinery.

Following the sample port integrity inspection, leachate sampling will commence using the following procedures:

- 1) The sample bottle shuttle from the laboratory will be opened and inspected to ensure that all the required sample containers are present, labeled, and contain preservatives, as necessary.
- 2) Active exhausts from combustion engines (vehicles, generators, etc.) will be placed clear of dry vegetation and downwind of the well to minimize cross-contamination.
- 3) Lay plastic sheeting around the port to minimize contamination of equipment from soil adjacent to the well.
- 4) Remove the lock and sample port cap and screen the headspace with a PID.

- 5) The pump will be started at a low flow rate (0.2 to 0.5 liters per minute) and will be slowly increased in speed. For samples collected for VOC analyses, the pump tubing needs to be completely filled with groundwater to minimize sample aeration as it flows through the tubing. Parameters which require field filtering will be collected last.
- 6) Field duplicates will be collected per the task-specific sampling areas. The sampler will alternate between filling an actual groundwater sample bottle for a given analysis and filling the associated duplicate bottle for the same analysis, with the exception of VOC which will have each bottle filled completely before alternating to another sampling bottle.
- 7) When field filtering is necessary (i.e., if turbidity measurements exceed 10 NTUs), the discharge line of the bladder pump will be fitted with a dedicated 0.45 micron (μm) in-line disposable filter. The filter will be rinsed following the manufacturer instructions, or if there are no instructions, a minimum of 0.5 to 1 liter of groundwater should pass through the filter before the sample is collected. The filtered sample will be directed into a laboratory prepared acid preserved bottle. If the sampling flow rate is not sufficient to push the water through the new filter, the groundwater will be pumped without a filter into a non-preserved, clean sample container. The bladder pump flow rate will not be changed for the purpose of using an in-line filter. If pumping with the bladder pump through the filter at existing flow rates is impeded by a clogged filter, a new filter may be installed and rinsed to complete the sample collection process. Alternately, the sample can be designated for laboratory filtering, followed by preservation. In this instance, the laboratory bottle will have no preservative added from the field.
- 8) The sample bottles will be labeled with sample identification, date, sample collection time, name of sampler, analyses requested, and any other pertinent information (e.g., filtered or unfiltered metals). The samples will then be placed in the sample cooler and chilled with ice to $\leq 6^{\circ}\text{C}$ or preserved as applicable to the specific analyses. A laboratory-provided temperature blank should be included in every sample cooler. The cooler will then be closed until the next sampling location is reached.
- 9) The field sampling record and the C-O-C form should be completed prior to mobilization to the next sampling location. If dedicated, the tubing of each well will be stored in its own individual, labeled plastic bag for future use, or will be disposed of at the Refinery's solid waste collection points. Dedicated tubing will not be stored in the well.
- 10) The monitoring well will be capped and locked following sampling, and all trash, tools, equipment, and decontamination and purge water will be removed from the area.
- 11) At the end of the day, the sample containers will be packed in the sample cooler in a manner to inhibit breakage. Additional ice will be added as needed to ensure that the samples remain chilled until arrival at the laboratory. The completed C-O-C form will be placed in a sealable watertight plastic bag to protect it from moisture and will be placed in the cooler or provided to the laboratory courier if transported directly to the laboratory by the courier. Custody seals will be placed on the cooler, and the cooler will be sealed with packing tape if it is being sent via overnight carrier. The cooler will be remanded to the overnight carrier or laboratory carrier for delivery to the laboratory.

4.4.4 Sediment Sampling

Sediment sampling locations will be identified in task-specific work plans prior to beginning field activities. Care will be taken to obtain as representative a sample as possible since samples will be collected from areas likely to be depositional environments. In addition to the generic equipment listed in Section 4.1, sampling equipment for sediment media are listed below.

- Trowels, hand tools, shovels, etc.;
- Decontamination equipment;
- Core Sampler;
- Bottom Grab sample such as a Ponar® Grab;
- Electric- or hand-powered boat and anchor (if performing surface water sampling on body of water), to be brought onsite and not stored at the Refinery;
- Life vests and water craft safety equipment.

Sediment samples will be collected by use of a decontaminated Ponar® Grab or other appropriate sampler, and the sediment transferred into a decontaminated, stainless-steel mixing bowl or disposable pan, if appropriate. Excess water will be decanted from the sample. Sediments for VOC analyses will be sampled immediately and placed into appropriate containers. Remaining sediments will be gently homogenized with a decontaminated stainless-steel spoon or disposable trowel and transferred into appropriate sampling containers. Sample containers should be completely filled. If sample material is limited, the Project Manager and the laboratory should be contacted to determine the minimum quantity required. Lid threads should be cleaned prior to closing the sample container to prevent potential contaminant migration to or from the sample and container leakage during shipment. Sample containers will be closed as soon as they are filled, chilled to $\leq 6^{\circ}\text{C}$, if appropriate, and processed for shipment to the laboratory.

To prevent cross-contamination of samples, boats used during sampling shall not be gasoline powered. The boat may be electric-powered or hand-powered. The boat shall be cleaned prior to use. Sampling shall begin at the most downstream sample location and proceed upstream to minimize water turbidity. Tidal and other currents should be considered when determining the upstream and downstream order of sampling.

The use of tripod drilling techniques from a pontoon boat may be required for borings to collect sediment in ponds or basins. With this method, a tripod and cathead are utilized to drive steel casing. The procedure is similar to using hollow-stem augers. The casing is advanced to the depth specified for sample collection. The sediment that has accumulated in the steel casing during advancement is washed out with water-flushed drilling rods. After the casing is washed out, the 2-inch diameter split-barrel sampler is lowered through the casing to depth and driven into the underlying sediment. If geotechnical data is desired, a 140-lb. hammer will be used to obtain the sample and blow counts will be recorded for each 6-inch increment the sampler is driven to assist in the assessment of the physical conditions of the sediment. When the sample is retrieved, it will be visually inspected for lithologic field classification and screened for VOCs using a PID. During sampling for composite samples, if an adequate sample volume cannot be collected from the initial boring, a subsequent boring located within approximately one foot of the original boring location can be drilled to collect the balance of the composite sample. This must be noted in the field log book.

4.4.5 Surface Water and Stormwater

Surface water and stormwater sampling locations will be identified in task-specific work plans prior to beginning field activities. Care will be taken to obtain as representative a sample as possible. In addition to the generic equipment listed in Section 4.1, sampling equipment for surface water or stormwater media are listed below.

- Bailers or dippers;
- Composite Liquid Waste Sampler (Coliwasa);
- Electric-or hand-powered boat and anchor (if performing surface water sampling on body of water), to be brought onsite and not stored at the Refinery;
- Life vests and watercraft safety equipment.

Surface-water samples will be collected using the dip method, where a decontaminated container is manually lowered into the body of water. Sample containers should be lowered upside down to the designated sample depth to prevent water from shallower depths from entering the container. The sample container is then inverted to allow water to enter and brought to the surface when filled or capped at depth and then brought to the surface. Samples can also be collected by lowering a capped container to the desired sample depth, uncapping the container and recapping it once the container is filled. Care should be taken to minimize turbulence caused by escaping air when collecting sample for volatile analyses. The filled container will be brought to the surface and the water transferred to laboratory-supplied containers. Sample containers should be completely filled. If sample material is limited, the PM and the laboratory should be contacted to determine the minimum quantity required. Lid threads should be cleaned prior to closing the sample container to prevent potential contaminant migration to or from the sample and to prevent container leakage during shipment. Samples collected for VOC analyses should be filled completely to the top, with zero headspace and no bubbles visible. Sample containers will be closed as soon as they are filled, chilled to $\leq 6^{\circ}\text{C}$, if appropriate, and processed for shipment to the laboratory.

To prevent cross-contamination of samples, boats used during sampling shall not be gasoline-powered. The boat may be electric powered or hand powered. The boat shall be cleaned prior to use. All sampling shall begin at the most downstream sample location and proceed upstream to minimize water turbidity. Tidal and other currents should be considered when determining the upstream and downstream order of sampling.

4.4.6 Groundwater

This section describes the procedures for gauging water levels, purging wells, and collecting groundwater samples. In addition to the generic equipment listed in Section 4.1, groundwater sampling equipment listed below is required for the sample event:

- Bladder pump or submersible pump and control box;
- Battery or gasoline powered generator for source of electric power;
- Multi-parameter water-quality meter with flow through cell and calibration kits;
- Water Level Meter and/or Interface Probe, as appropriate;
- Dedicated Teflon™-lined polyethylene tubing;
- Dedicated Teflon™-lined bladders;

- Containers for containerizing purge water and equipment cleaning fluids;
- Paper towels, trash bags, small plastic bags, packing tape;
- Measuring tape and zip ties;
- Keys for well locks;
- Bolt cutters.

Water-Level Measurements

Field-meter probes will be cleaned before and after use at each well. Prior to any field measurements, the integrity of each well shall be inspected. If there is any reason that field measurements or samples cannot be obtained from a well, the reason should be noted in the field log book. The field team shall notify the Project Manager who will notify the Project Coordinator and the DNREC Case Manager.

Total volatile organic readings will be measured with a PID immediately after opening each well. PID data will be recorded in the field records. Synoptic water-level measurements and total well-depth measurements will be taken at all wells in the sampling program prior to any well purging or groundwater sampling. An interface probe, accurate to 0.01 feet, will be used to measure depth-to-water to the nearest 0.01 feet in each well. When using an electronic meter, the probe is lowered down the well casing to the top of the water column; the graduated markings on the probe wire or tape are used to measure the depth-to-water from the marked surveyed point on the rim of the inner well casing, not the stickup protective casing or flush-mount box. Typically, the measuring device emits a constant tone when the probe is submerged in standing water and most electronic water level sounders have a visual indicator consisting of a small light bulb or diode that turns on when the probe encounters water. Total well depth will be measured to the nearest 0.01 feet from the top of the well casing by lowering the weighted probe to the bottom of the well. Care should be taken to minimize disturbing sediments which may have accumulated at the bottom of the well. If portions of the water level meter or interface probe are present below the zero point of the measuring tape, the length of the meter should be determined and added to all well depth measurements.

Water-level sounding equipment will be cleaned before use in each well. Water levels will be measured in wells that have no known contamination or the least amount of known contamination first, followed by wells with anticipated higher levels of contamination. However, as it is critical to obtain synoptic water levels from all wells as rapidly as practicable; wells of varying impact may be measured at random in order to move swiftly across an area.

Purging

Wells will be purged prior to collecting groundwater samples. Low-flow sampling and bladder pumps will be the primary well purging and groundwater sampling pumps used at the Refinery. The depth-to-water, water-quality parameter measurements, and purge volumes will be entered in the logbook and/or field data sheets. The water in which stabilization parameters were measured will not be used to fill sample bottles.

The following procedures will be followed prior to collecting groundwater samples:

- 1) The well volume will be calculated based on the well casing diameter and using the depth to the bottom of the well and the depth-to-water measurements collected during the synoptic water level event conducted at the initiation of the groundwater sampling program.

- 2) Unless the monitoring well has a dedicated pump, the type of groundwater pump to be used (such as bladder or submersible pump) will be determined by well depth. Generally, bladder pumps are used up to depths of approximately 90 to 100 feet bgs and submersible pumps are used at depths greater than 100 feet bgs. The selected pump should be capable of maintaining low flow rates with minimal disturbance to the water column.
- 3) A new or dedicated Teflon™ bladder will be installed in the bladder pump prior to sampling each well. New or dedicated Teflon™-lined or polyethylene tubing will be attached to the pump. A clean tape measure will be attached to the pump, using zip ties so that the pump intake can be set at the proper depth. Other methods of determining the pump intake depth may be utilized upon approval of the Project Manager.
- 4) The pump should be lowered slowly to the target pump intake depth (discussed below) to minimize disturbance of the water column and sediment. While the pump is being lowered into the well, the tubing and tape measure should be wiped with a paper towel saturated with distilled water. Care will be taken not to lower the pump past the targeted intake depth to avoid disturbing sediments that may have settled at the bottom of the well.
- 5) The depth of the pump intake will be determined based on the length of the well screen, lithologic characteristics, or suspected zones of contamination. The pump intake will be positioned at the point within the screened interval where the suspected highest concentration of potential contaminants is located, or, if not known, the middle of the screened interval. If there are no targeted zones of suspected contamination and the screen extends above the water table, the pump will be set at half the distance between the top of the static water column and the bottom of the well screen.
- 6) A depth-to-water meter will be lowered into the well to monitor the water column drawdown during purging and sampling. The line of the depth-to-water meter should be wiped with a towel saturated with distilled water during insertion into the well.
- 7) The discharge tubing will be connected to a flow-through-cell water-quality parameter meter to monitor water-quality parameters and to determine when they have stabilized. A discharge line should also be attached to the flow-through cell and directed to a container for the collection of purge water.
- 8) The pump will be started at a low flow rate (0.2 to 0.5 liters per minute) and will be slowly increased in speed. The water level will be monitored to determine an appropriate flow rate to maintain a water-column drawdown of less than 0.33 feet below the original pre-pump installation static water level. If the drawdown is, or predicted to be, greater than 0.33 feet, the flow rate will be reduced. A drawdown of less than 0.33 feet is the goal for determining the flow rate and may be difficult to achieve due to the lithology or other factors. Therefore, well-specific adjustments to the flow rate may be required.

For double-screened wells, the above procedures will be followed; however, each screened interval is considered to be separate and should be treated as such. In cases where historical well data are available, the screened interval with the least amount of contamination will be sampled first. In cases where the concentrations of contaminants are unknown, the uppermost screened interval will be sampled first.

The following rationale for wells with screens greater than 20 feet will be used:

Well ID	Screened Interval (feet bgs)	Placement of Bladder Pump (feet bgs)	Rationale for Pump Placement
D7	28-57	38 and 48	Sample at $\frac{1}{3}$ and $\frac{2}{3}$ of screen
D9	16-46	20 and 30	Sample at $\frac{1}{3}$ and $\frac{2}{3}$ of screen
C31	32-72	45 and 58	Sample at $\frac{1}{3}$ and $\frac{2}{3}$ of screen

Water-quality measurement readings will be recorded continually while purging from time zero (0) using a water-quality measurement instrument equipped with a flow through cell. Water-quality measurements will begin once one volume of water has been purged through the flow thru cell, the tubing, and the bladder. The time it takes for one volume to pass through is calculated using the following formula:

$$\text{Time (min)} = \frac{(t_{ft}) (9.8004 \text{ cm}^3) + 500 \text{ ml}}{\text{Purge rate (ml/min)}}$$

Where: t_{ft} is the length of tubing in feet

9.8004 is a constant calculated by multiplying the following:

Interior radius of tubing (0.32 cm) squared (0.1024 cm^2) x pi (3.14) x 30.48 cm (number of cm in 1 ft)

Volume of water at any one time in bladder = 100 ml

Volume of water at any one time in flow thru cell = 400 ml

Water-quality parameters will be compared to the criteria presented below. Parameters have reached stabilization when three consecutive readings (collected at five-minute intervals) for all parameters are below the requirements. Well purge is complete when water-quality parameters have stabilized. If water-quality standards have not stabilized after six (6) consecutive readings or within 30 minutes, then purging will cease and sampling will begin. This will be noted in the field log book.

To obtain a groundwater sample that is representative of surrounding aquifer conditions, stable water-quality parameters (temperature, pH, specific conductance, ORP, turbidity, and dissolved oxygen) measurements should be obtained, if possible. During purging, water-level drawdown in the well will be maintained at less than 0.33 feet drawdown, if reasonable. Water-quality parameters are considered stable if the following criteria are achieved for three consecutive readings:

Temperature range is no more than $\pm 3\%$;

pH varies by no more than ± 0.1 pH units;

Specific conductance readings are within $\pm 3\%$;

Oxidation Reduction Potential (ORP) is ± 10 millivolts;

Turbidity is $\pm 10\%$ NTU (when turbidity is greater than 10 NTU); and

Dissolved oxygen is $\pm 10\%$ when greater than 0.5 mg/L or ± 0.1 mg/L when less than 0.5 mg/L.

Submersible (or centrifugal) pumps may also be used as an alternative to bladder pumps upon approval of the Project Manager and the DNREC Case Manager. If submersible pumps are used, then water-quality parameters will be recorded every five minutes. Parameters have reached stabilization when three consecutive readings for all parameters are below the requirements. If water-quality standards have not stabilized after six (6) consecutive readings or within 30 minutes, then purging will cease and sampling will begin. This will be noted in the field log book. Under some conditions including, analyte lists (e.g. no VOC analyses), the diameter and/or configuration of the well, well damage, depth-to-water, etc., other pumps such as a peristaltic pump may be utilized upon approval of the Project Manager.

Clean, flexible Teflon™-lined polyethylene tubing will be used for groundwater extraction. Dedicated tubing will be used for each well to prevent cross-contamination. Pumps will be placed at depths within the screened interval where the suspected highest concentration of potential contaminants is located, or, if not known, the middle of the screened interval as determined via the procedures described above.

Water will be collected into a graduated container to record the purge volume. Casing volumes will be calculated based on total well depth, static water level, and casing/screen diameter. One casing volume will be calculated as:

$$V = \pi d^2 h / 77.01$$

Where:

V is the volume of one well casing of water ($1 \text{ ft}^3 = 7.48$ gallons);

d is the inner diameter of the well casing (in inches);

h is the total depth of water in the well (in feet).

Note: A 2-inch ID well contains 0.16 gallons of water per foot. A 4-inch ID well contains 0.65 gallons of water per foot.

If a well dewateres (runs dry) during purging and either the parameters have not stabilized, or five casing volumes have not been purged, that well will be allowed to recharge to 80% of the static water column. After the water level has recharged to 80% of the static water column, groundwater samples will be collected

A well is allowed to be dewatered after both of the following steps are taken:

- 1) If after a pumping rate has been established, the drawdown exceeds 0.33 feet, reduce the pump flow until drawdown has stabilized.
- 2) If drawdown has not stabilized and the flow rate is under 100 milliliters per minute.

Permanent Monitoring Well Sampling

Sampling, sample handling, and decontamination will be conducted with appropriate PPE based on the constituents known to exist in the groundwater (based on historical groundwater data or refinery process data, where available). Purge water and decontamination water will be containerized and transported to the Refinery

WWTP for disposal upon approval by the WWTP Operator. Water will not be discharged onto the ground surface unless prior approval is given by the Refinery.

Following the well-integrity inspection and depth-to-water measurements at all wells, groundwater sampling will commence using the following procedures:

- 1) The sample bottle shuttle from the laboratory will be opened and inspected to ensure that all the required sample containers are present, labeled, and contain preservatives, as necessary.
- 2) Sampling will begin at the well that historically has been the generally least contaminated or is believed to be the least contaminated based on historical analytical data, its location as the most upgradient well, or the well farthest from a suspected source area. The wells will be sampled systematically from generally the least contaminated to the most contaminated.
- 3) Active exhausts from combustion engines (vehicles, generators, etc.) will be placed clear of dry vegetation and downwind of the well to minimize cross-contamination.
- 4) Lay plastic sheeting around the well to minimize contamination of equipment from soil adjacent to the well.
- 5) Remove the locking and well casing caps and screen the headspace of the well with a PID.
- 6) Purging of the wells will begin as described in the previous section. Once field parameters have stabilized, a final reading will be taken of all of the parameters.
- 7) Prior to sampling, the same pumping rate will be maintained or reduced slightly to minimize disturbance to the water column during sampling. For samples collected for VOC analyses, the pump tubing needs to be completely filled with groundwater to minimize sample aeration as it flows through the tubing. Parameters which require field filtering will be collected last.
- 8) Field duplicates will be collected per the task-specific sampling areas. The sampler will alternate between filling an actual groundwater sample bottle for a given analysis and filling the associated duplicate bottle for the same analysis, with the exception of VOC which will have each bottle filled completely before alternating to another sampling bottle.
- 9) Sample bottles will be filled in the following order: VOCs, gasoline range organics, total organic carbon, semivolatile organic compounds, diesel range organics, total petroleum hydrocarbons / oil and grease, polychlorinated biphenyls, pesticides, sulfate and chloride, nitrate and ammonia, preserved inorganics, non-preserved inorganics, bacteria, total metals (unfiltered), and dissolved metals (filtered).
- 10) When field filtering is necessary (i.e., if turbidity measurements exceed 10 NTUs), the discharge line of the bladder pump will be fitted with a dedicated 0.45 micron (μm) in-line disposable filter. The filter will be rinsed following the manufacturer instructions, or if there are no instructions, a minimum of 0.5 to 1 liter of groundwater should pass through the filter before the sample is collected. The filtered sample will be directed into a laboratory prepared acid preserved bottle. If the sampling flow rate is not sufficient to push the water through the new filter, the groundwater will be pumped without a filter into a non-preserved, clean sample container. A peristaltic pump using dedicated tubing will then be used to pump the sample through the filter into the sample container that will be submitted to the lab. The bladder pump flow rate will not be changed for the purpose of using an in-line filter. If pumping with the bladder pump through the filter at

existing flow rates is impeded by a clogged filter, a new filter may be installed and rinsed to complete the sample collection process. Alternately, the sample can be designated for laboratory filtering, followed by preservation. In this instance, the laboratory bottle will have no preservative added from the field.

- 11) The sample bottles will be labeled with sample identification, date, sample collection time, name of sampler, analyses requested, and any other pertinent information (e.g., filtered or unfiltered metals). The samples will then be placed in the sample cooler and chilled with ice to $\leq 6^{\circ}\text{C}$ or preserved as applicable to the specific analyses. A laboratory-provided temperature blank should be included in every sample cooler. The cooler will then be closed until the next sampling location is reached.
- 12) The field sampling record and the C-O-C form should be completed prior to mobilization to the next sampling location. If dedicated, the tubing of each well will be stored in its own individual, labeled plastic bag for future use, or will be disposed of at the Refinery's solid waste collection points. Dedicated tubing will not be stored in the well.
- 13) The monitoring well will be capped and locked following sampling, and all trash, tools, equipment, and decontamination and purge water will be removed from the area.
- 14) At the end of the day, the sample containers will be packed in the sample cooler in a manner to inhibit breakage. Additional ice will be added as needed to ensure that the samples remain chilled until arrival at the laboratory. The completed C-O-C form will be placed in a sealable watertight plastic bag to protect it from moisture and will be placed in the cooler or provided to the laboratory courier if transported directly to the laboratory by the courier. Custody seals will be placed on the cooler, and the cooler will be sealed with packing tape if it is being sent via overnight carrier. The cooler will be remanded to the overnight carrier or laboratory carrier for delivery to the laboratory.
- 15) For double-screened wells, the above procedures will be followed; however, each screened interval is considered to be separate and should be treated as such. In cases where historical well data are available, the screened interval with the least amount of contamination will be sampled first. In cases where the concentrations of contaminants are unknown, the uppermost screened interval will be sampled first.

Temporary Monitoring Well Sampling

Direct-push drilling techniques (e.g., Geoprobe[®]) can be used to install temporary monitoring wells for the collection of groundwater samples. The preferred method for temporary monitoring well installation is the traditional filter pack method. However, the specific installation procedure will be specified in the work plan and submitted to DNREC for approval.

Due to the smaller diameter of temporary wells, peristaltic pumps with disposable tubing will be used to develop the wells and collect groundwater samples. Temporary monitoring wells will be developed by pumping until the evacuated groundwater visually clears, or as otherwise specified in the task-specific workplan. After development of temporary monitoring wells, groundwater field parameters (pH, specific conductance, temperature, dissolved oxygen, and turbidity) will be measured and documented in the field notes. Groundwater samples will be collected with a peristaltic pump, preferentially using low-flow sampling techniques, for parameters specified in the task-specific work plans.

4.5 Field Quality Control Samples

Field QC samples require prior planning to determine the frequency (number) and type (blanks, spikes) to be sent from the field to the laboratory. Field QC samples are not used to monitor the variations in the laboratory, but, rather, field conditions that may affect the results of the actual samples. In the laboratory, field QC samples are processed as any other field sample.

4.5.1 Assessment of Field Contamination

Field contamination is assessed using equipment and trip (VOCs only) blanks.

Equipment Blanks

Equipment rinsate blanks will be collected to evaluate field sampling and decontamination procedures by pouring deionized water, supplied by the laboratory, over decontaminated sampling equipment. One equipment rinsate blank will be collected per matrix each day or at a frequency of one equipment blank per 20 or fewer field samples that are collected in the field, whichever is more frequent. The rinsate blanks are analyzed for the same parameters requested for the associated field samples. The same types of bottles should be used for equipment blanks as are being used for field samples.

The equipment rinsate blanks will be preserved, packaged, and sealed in the same manner described for the environmental samples. A separate sample number will be assigned to each sample. Equipment blanks will use the following nomenclature:

EB-followed by the date

If more than one equipment blank is collected during the same day, each equipment blank will have an increasing number assigned after the “EB” (i.e., EB1-(date), EB2-(date), etc.).

Trip Blanks

Trip blanks will be prepared to evaluate if the shipping and handling procedures are introducing contaminants into the samples, and if cross contamination in the form of VOC migration has occurred between the collected samples. A minimum of one trip blank per cooler containing aqueous samples for VOC analyses will be submitted to the laboratory. Trip blanks are 40 ml vials that have been filled by the laboratory with organic-free, deionized water and shipped with the empty coolers to the site prior to sampling. The sealed trip blanks are not opened in the field and are shipped to the laboratory in the same cooler with the samples collected for VOC analyses. The trip blanks will be preserved, packaged, and sealed in the manner described for the environmental samples. A separate sample number will be assigned to each sample. Trip blanks will use the following nomenclature:

TB-followed by the date

If more than one trip blank is collected during the same day, each trip blank will have an increasing number assigned after the “TB” (i.e., TB1-(date), TB2-(date), etc.).

4.5.2 Assessment of Field Variability (Field Duplicates)

Field duplicate samples are collected to compare field sampling variability. Field duplicates are collected simultaneously with a standard sample from the same source under identical conditions into separate sample containers. Each field duplicate will be labeled as “FD”, assigned a separate sample number, and sent as a blind sample to the laboratory. At least 5% (one per 20 field samples) of all field samples collected per sampling event

should be field duplicates. At least one duplicate sample per sample matrix should be collected per sampling event; however, their collection can be stretched out over more than one day.

Field duplicate samples will be preserved, packaged, and sealed in the same manner as the environmental samples. Field duplicate samples will use the following nomenclature:

FD-followed by the date

If more than one field duplicate sample is collected during the same day, each duplicate will have an increasing number assigned after the "FD" (i.e., FD1-(date), FD2-(date), etc.). The field sample for which the duplicate sample is associated must be noted on the field data sheet and in the field log book at the time of collection.

4.5.3 Assessment of Matrix Effects

Matrix effects are evaluated through the laboratory analysis of matrix spike/matrix spike duplicate (MS/MSD) samples. Extra sample volume is collected in the field to provide the laboratory with sufficient material to perform MS/MSD analyses. At least 5% (one per 20 field samples) of all field samples collected per sampling event should have extra sample volume collected for MS/MSD analyses. Extra volume collected for laboratory QA/QC samples such as MS/MSD will be noted as a comment on the C-O-C form. MS/MSD samples are not entered separately as field samples on the C-O-C form.

4.6 Decontamination

The decontamination procedures for field sampling equipment are described below and will be strictly adhered to.

4.6.1 Surface/Sediment/Subsurface Soil Sampling Equipment

Sampling equipment will be constructed of inert materials and if not discarded, will be decontaminated in the field prior to use, between each sample, and at the completion of the sampling program. The decontamination method will consist of the following steps:

- 1) Non-phosphate detergent and tap water wash;
- 2) Tap water rinse; and
- 3) Distilled- or deionized-water rinse.

In addition, the drilling contractor will use a high pressure, hot-water wash, and the field geologist/engineer will confirm by visual inspection that the tools and any downhole components or materials have been hot-water washed immediately before work begins and between each boring location. An on-site controlled decontamination area will be selected for equipment cleaning. Specific decontamination procedures for the drill rig and drilling equipment will consist of:

- 1) High pressure, hot-water wash all equipment that will be introduced into the borehole.
- 2) Storing all augers and equipment used in drilling off of ground surfaces and/or on clean plastic until they are required for use.
- 3) Use dedicated latex/nitrile gloves when handling sampling equipment. Gloves and other PPE will be collected in garbage bags and disposed of at the conclusion of daily operations.

These decontamination procedures will be employed for all drilling activities. Their purpose is to minimize the potential for transferring possible contaminants from one borehole to another.

4.6.2 Groundwater Equipment

The water-level meter and any reusable equipment contacting the well will be cleaned in the following manner:

- 1) Wipe with a paper towel soaked with either distilled or deionized water and non-phosphate detergent to remove visible contamination, if present.
- 2) Where practical, hand wash with a brush in tap water and non-phosphate detergent.
- 3) Rinse with tap water.
- 4) Rinse with distilled or deionized water.

The bladder pump will be cleaned in the following manner:

- 1) Wipe with a paper towel to remove visible contamination, if present.
- 2) Remove and discard associated disposable fittings from the pump.
- 3) Hand wash with a brush in tap water and non-phosphate detergent.
- 4) Rinse with tap water.
- 5) Rinse with distilled or deionized water.
- 6) Install a new bladder in the pump.
- 7) Dedicated bladders are cleaned and placed in a ziplock bag and properly identified.

A submersible pump will be cleaned in the following manner:

- 1) Pump and lead are placed into a five-gallon bucket of tap water and non-phosphate detergent and allowed to run for three minutes. The wash water is recirculated back into the bucket.
- 2) Pump and lead are removed from detergent bucket and placed into a tap water rinse and allowed to run for an additional three minutes. The rinse water is recirculated back into the bucket.
- 3) The lead is rolled onto the spindle through a distilled water-soaked paper towel.

The detergent water and tap water should be changed at least once a day or when water becomes visibly dirty. Pump decontamination procedures may need to be modified if a pump other than a bladder or submersible pump is used for sampling.

5.0 SAMPLE CONTAINERS, PRESERVATION AND ANALYSIS

The number of samples containers, volumes, and materials are listed in Table 1. The containers are pre-cleaned and will not be rinsed prior to sample collection. Preservatives, if required, will be added by the laboratory during bottle order preparation. Sample volume may be reduced for samples containing elevated analyte concentrations or for waste samples. Sample volumes or containers may be reduced as laboratories develop low sample volume

analytical methods, provided the methods meet the method quantitation limits specified in the task-specific work plan and the precision and accuracy requirements in the QAPjP.

6.0 SAMPLE DOCUMENTATION AND SHIPPING

This section describes the field procedures for log books/sampling forms, labeling, C-O-C forms, custody seals, sample packaging, and shipping.

6.1 Field Log Books/Field Sampling Forms

An example field data sheet is provided as Figure 1. At a minimum, the following information will be recorded during the collection of each sample:

- Site (waste unit) name and project number
- Name of contractor, subcontractor, and visitor personnel on site, including name(s) and organization(s)
- Time of arrival (entering on site) and time of site departure
- Descriptions of site activities, including dates and times of logbook entries and site entry and exit times
- Noteworthy events and discussions, including summaries of any meetings or discussions with contractors or regulatory agencies
- Identification, location, and description of samples collected
- Site or sampling area sketch showing sample location and measured distances
- Sampler's name(s)
- Date and time of sample collection
- Type of sample collected
- Type of sampling equipment used
- Field instrument readings and calibration data
- Field observations and details relating to analysis or integrity of samples (i.e., weather conditions, noticeable odors, colors, etc.)
- Deviations from sampling plans, site safety plans, and QAPjP procedures
- Changes in personnel and responsibilities with reasons for changes
- Levels of safety protection (i.e., PPE)
- Designation of sample as composite or grab
- Type of sample (soil, sediment, leachate, or water)
- Preliminary sample descriptions (i.e., for soil – clay loam, very wet; for water – clear with strong hydrocarbon odor)

- Calibration readings for any equipment used, and equipment model and serial number

6.2 Labeling

Sample bottles will be labeled in a clear and unique manner for proper identification in the field and for tracking in the laboratory. An example sample bottle label is provided in Figure 2. The samples will have pre-assigned unique numeric or alpha-numeric identification numbers. The sample bottles will be labeled with sample identification, date, sample collection time, name of sampler, analyses requested, and any other pertinent information.

Bottles will be ordered from the laboratory and inspected prior to any field activities.

6.3 Sample Chain-of-Custody Forms and Custody Seals

Sample shipments to the laboratory will be accompanied by a C-O-C form. An example form is provided as Figure 3. C-O-C form(s) will be completed and sent with the samples for each shipment. If multiple coolers are sent to a single laboratory on a single day, C-O-C form(s) will be completed and sent with the samples in one of the coolers.

The C-O-C form will identify the contents of each shipment and document the custodial integrity of the samples. Generally, a sample is considered to be in someone's custody if it is either in someone's physical possession, in someone's view, locked up, or kept in a secure area with restricted access to authorized personnel. At the time of shipment, the sampling team leader or designee will sign the C-O-C form in the "relinquished by" box and note date, time, and, if being shipped by common carrier, the air bill number.

The C-O-C form will be filled out in ink. The C-O-C form will indicate the following information:

- Project name;
- Unique sample number;
- Sample matrix;
- Number of bottles for each sample and analyte;
- Date of collection;
- Time of collection;
- Analysis requested;
- Preservatives used;
- Sampler's name;
- Sampler's signature and date;
- Sample bottle order number (if provided); and
- If appropriate, notes on unusual sample characteristics that may be important to laboratory personnel, such as elevated PID readings, suspected high contaminant concentrations, evidence of off-gassing, etc.

The sample numbers for all QC samples including rinsate blanks, trip blanks, duplicate samples, reference samples, and laboratory QA samples (MS/MSD samples) will be documented on the C-O-C form. Once samples are packed in the cooler, the pink copy of the C-O-C form is retained by the sampling team leader. The C-O-C form is scanned into PDF and filed electronically. The remainder of the C-O-C form is then placed in a ziplock bag and placed inside the cooler.

An example custody seal is provided as Figure 4. The shipping containers in which samples are stored (i.e., coolers) will be packed with ice or an approved alternate (i.e., blue ice), and will be sealed with self-adhesive custody seals and packing tape when the samples are not in custody as defined above.

6.4 Packaging and Shipment

The following outlines the packaging procedures that will be followed for all samples:

- 1) Seal the drain plug of the cooler to prevent melting ice from leaking out of the cooler.
- 2) Line the bottom of the cooler with bubble wrap to prevent breakage during shipment.
- 3) Check screw caps for tightness.
- 4) Secure bottle/container tops with clear tape.
- 5) Affix sample labels onto the containers with clear tape, if needed.
- 6) Wrap all glass sample containers in bubble wrap to prevent breakage.
- 7) Place samples in a sturdy cooler lined with a large plastic trash bag. Enclose the appropriate C-O-C forms in a sealable watertight plastic bag affixed to the underside of the cooler lid.
- 8) For each cooler that is shipped or transported to an analytical laboratory, a container will be included that is marked "temperature blank". This blank will be used by the sample custodian to check the temperature of samples upon arrival to the laboratory. The temperature of each cooler will be recorded on the C-O-C. The temperature blank is not listed on the C-O-C form and will not be analyzed.
- 9) Ice used to cool samples will be sealed in sealable watertight plastic bags and placed on top and around the samples to chill them to the correct temperature.
- 10) Each cooler will be securely taped shut with packing tape, and custody seals will be affixed to the front, right, and back of the cooler. The coolers will be labeled with appropriate DOT shipping labels.
- 11) Affix the correct shipping label (either next day service or Saturday service) to the top of the cooler. It is important to inform the laboratory if shipping samples for Saturday delivery.

7.0 IDW DISPOSAL

In the process of collecting environmental samples at the Refinery, the sampling team will generate different types of potentially contaminated waste including the following:

- Used PPE;
- Disposable sampling equipment;

- Decontamination fluids;
- Soil cuttings from soil borings;
- Purged groundwater and excess groundwater collected from sample-container filling.

This waste will be handled in accordance with the following protocols:

- Used PPE and disposable sampling equipment will be bagged and placed in a municipal refuse dumpster. These wastes are not considered hazardous and can be sent to a municipal landfill.
- Decontamination fluids that will be generated during field sampling efforts will consist of deionized or distilled water, residual contaminants, and water with non-phosphate detergent. These fluids, along with any purged groundwater, will be transported to the WWTP for proper disposal upon approval from the WWTP. If the WWTP does not accept the fluids, fluids will be contained in labeled DOT-approved 55-gallon drums and disposed of at an appropriate offsite waste disposal facility.
- Soil cuttings, drilling muds, and excess grout generated during the subsurface sampling will be placed into either lined or covered roll-offs or DOT-approved 55-gallon drums for offsite disposal at an approved waste disposal facility. Prior to solid or liquid disposal, the material will be sampled and analyzed for parameters defined by the waste disposal facility for appropriate characterization.

8.0 FIELD VARIANCES

As conditions in the field may vary, it may become necessary to implement minor modifications to sampling as presented in this plan. The Project Manager or the Quality Assurance Officer should be notified and a verbal approval should be attained prior to implementing the changes. Modifications to the approved plan will be documented in the field notes as well as the sampling project report. Modifications other than minor will require DNREC approval prior to implementation.

9.0 FIELD HEALTH AND SAFETY PROCEDURES

Health and safety procedures are to be followed using the most recently updated Delaware City Refinery HASP. Any changes or deviations from the HASP must be approved by the Project Manager and/or Health and Safety Officer prior to implementing the change. Changes to the HASP shall be documented in the field notes.

10.0 REFERENCES

American Public Health Association (APHA). Standard Methods for the Examination of Water and Wastewater; <https://www.standardmethods.org/>

ASTM – ASTM International; <https://www.astm.org/Standards>

Department of Natural Resources and Environmental Control (DNREC). 2016. Site Investigation and Restoration Section Standard Operating Procedure Active Soil Gas Sampling Procedure. Updated June 24, 2016. <http://www.dnrec.delaware.gov/dwhs/SIRB/Documents/9-16-16%20Active%20Soil%20SOP.pdf>

URS Corporation (URS). 2011. Phase II RCRA Facility Investigation Groundwater. May 2011

U. S. Environmental Protection Agency (USEPA). 1986. EPA Test Methods for Evaluating Solid Waste - Physical/Chemical Methods", EPA Publication SW-846. <https://www.epa.gov/hw-sw846/sw-846-compendium>.

U. S. Environmental Protection Agency (USEPA). 1999 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, EPA/625/R-96/010B;
<https://www3.epa.gov/ttnamti1/files/ambient/airtox/to-15r.pdf>

U. S. Environmental Protection Agency (USEPA). 2017. Methods for the Chemical Analysis of Water and Wastes, 82 FR 40846, Aug. 28, 2017 and 40 CFR 136. <https://www.epa.gov/cwa-methods/approved-cwa-chemical-test-methods>

Table 1: Analyses, Methods, Sample Containers, and Preservation/Holding Time Requirements

Analysis and Method	Matrix	Sample Container	Preservation	Holding Time
Volatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS) (SW-846 8260D)	Water	3 - 40ml vials	HCl to pH < 2; cool to ≤ 6°C	14 days from collection to analysis
Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS) (SW-846 8270E)	Water	2 - 1 Liter amber bottles	Cool to ≤ 6°C	7 days from collection to extraction; 40 days from extraction to analysis
Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (SW-846 6010D) or by Inductively Coupled Plasma-Mass Spectrometry (6020B)	Water	500 mL plastic	HNO ₃ to pH < 2	180 days from collection to analysis
Mercury in Liquid Waste (Manual Cold-Vapor Technique) (SW-846 7470A)	Water	100 mL plastic	HNO ₃ to pH < 2	28 days from collection to analysis
Total and Amenable Cyanide (Automated Colorimetric, with Off-Line Distillation (SW-846 9012B)	Water	250 mL plastic	NaOH to pH >12; ascorbic acid, cool to ≤ 6°C	14 days from collection to analysis
Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides (SW-846 9030B/9034)	Water	250 mL plastic	NaOH to pH >9; zinc acetate, cool to ≤ 6°C	7 days from collection to analysis
Total Organic Carbon (SW-846 9060A)	Water	250 mL plastic	HCl to pH < 2; cool to ≤ 6°C	28 days from collection to analysis
n-Hexane Extractable Material (HEM: Oil and Grease) (EPA 1664B)	Water	2 - 1 Liter glass bottles	HCl or H ₂ SO ₄ to pH < 2; cool to ≤ 6°C	28 days from collection to analysis
Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) (EPA 1664B)	Water	2 - 1 Liter glass bottles	HCl or H ₂ SO ₄ to pH ≤ 2; cool to ≤ 6°C	28 days from collection to analysis
Organochlorine Pesticides (SW-846 8081B)	Water	2 - 1 Liter amber bottles	Cool to ≤ 6°C	7 days from collection to extraction; 40 days from extraction to analysis
Polychlorinated Biphenyls (PCBs) by Gas Chromatography (SW-846 8082A)	Water	2 - 1 Liter amber bottles	Cool to ≤ 6°C	None

Analysis and Method	Matrix	Sample Container	Preservation	Holding Time
Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (EPA 1668C)	Water	2 – 1 Liter amber bottles	Store samples in dark; cool to $\leq 6^{\circ}\text{C}$ Store extracts in dark at $<-10^{\circ}\text{C}$	1 year from collection to extraction; 1 year from extraction to analysis
Polychlorinated Dibenzo-p-Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (SW-846 8290A)	Water	2 - 1 Liter amber bottles	Store in dark; cool to $\leq 6^{\circ}\text{C}$	None
Total Petroleum Hydrocarbons as Gasoline (Nonhalogenated Organics by Gas Chromatography, SW-846 8015C)	Water	2 - 40 mL vials	HCl to $\text{pH} < 2$; cool to $\leq 6^{\circ}\text{C}$	14 days from collection to analysis
Total Petroleum Hydrocarbons as Diesel (Nonhalogenated Organics by Gas Chromatography, SW-846 8015C)	Water	2 - 1 Liter amber bottles	Cool to $\leq 6^{\circ}\text{C}$	7 days from collection to extraction; 40 days from extraction to analysis
Determination of Inorganic Anions by Ion Chromatography (SW-846 9056A) (bromide, chloride, fluoride, nitrate, nitrite, phosphate or sulfate)	Water	125 mL plastic	Cool to $\leq 6^{\circ}\text{C}$	28 days from collection to analysis
Alkalinity, as CaCO_3 (SM 2320B)	Water	250 mL plastic	Cool to $\leq 6^{\circ}\text{C}$	14 days from collection to analysis
Biochemical Oxygen Demand (SM 5210B)	Water	1000 mL plastic	Cool to $\leq 6^{\circ}\text{C}$	48 hours from collection to analysis
Chemical Oxygen Demand by Semi-Automated Colorimetry (EPA 410.4)	Water	100 mL plastic	H_2SO_4 to $\text{pH} < 2$; cool to $\leq 6^{\circ}\text{C}$	28 days from collection to analysis
Phosphorus, Total, Colorimetric, Automated, Block Digester, Automated Analyzer II (EPA 365.4)	Water	250 mL plastic	H_2SO_4 to $\text{pH} < 2$; cool to $\leq 6^{\circ}\text{C}$	28 days from collection to analysis
Heterotrophic Plate Count (SM 9215-2017)	Water	125 mL plastic	Sodium Thiosulfate, Cool to $\leq 10^{\circ}\text{C}$	8 hours (recommended)/24 hours (maximum) from collection to analysis
Total Kjeldahl Nitrogen by Semi-Automated Colorimetry (EPA 351.2)	Water	125 mL plastic	H_2SO_4 to $\text{pH} < 2$; cool to $\leq 6^{\circ}\text{C}$	28 days from collection to analysis

Analysis and Method	Matrix	Sample Container	Preservation	Holding Time
Volatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS) (SW-846 5035/8260D)	Soil/ Sediment	3 Encore™ samples or 3 vial Terracore™ kit (2 water and 1 methanol) Do NOT add sample label to vials; vials are pre-weighed 2 oz. jar (sediment only)	Cool to ≤ 6°C	Encore™ or unpreserved Terracore™ samples - 48 hours from collection to extraction or freeze Preserved Terracore™ samples – 14 days from collection to analysis Sediment samples - 14 days from collection to analysis
Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS) (SW-846 8270E)	Soil/ Sediment	4 oz jar	Cool to ≤ 6°C	14 days from collection to extraction; 40 days from extraction to analysis
Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (SW-846 6010D) or by Inductively Coupled Plasma-Mass Spectrometry (6020B)	Soil/ Sediment	4 oz jar	None	180 days from collection to analysis
Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique) (SW-846 7471B)	Soil/ Sediment	4 oz jar	Cool to ≤ 6°C	28 days from collection to analysis
Total and Amenable Cyanide (Automated Colorimetric, with Off-Line Distillation (SW-846 9012B)	Soil/ Sediment	4 oz jar	Cool to ≤ 6°C	14 days from collection to analysis
Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides (SW-846 9030B/9034)	Soil/ Sediment	4 oz jar	Cool to ≤ 6°C	7 days from collection to analysis
Total Organic Carbon by Lloyd Kahn Method	Soil/ Sediment	4 oz jar	Cool to ≤ 6°C	28 days from collection to analysis
n-Hexane Extractable Material (HEM) for Sludge, Sediment, and Solid Samples (SW-846 9071B)	Soil/ Sediment	4 oz jar	Cool to ≤ 6°C	28 days from collection to analysis
Organochlorine Pesticides (SW-846 8081B)	Soil/ Sediment	4 oz jar	Cool to ≤ 6°C	14 days from collection to extraction; 40 days from extraction to analysis

Analysis and Method	Matrix	Sample Container	Preservation	Holding Time
Polychlorinated Biphenyls (PCBs) by Gas Chromatography (SW-846 8082A)	Soil/ Sediment	4 oz jar	Cool to $\leq 6^{\circ}\text{C}$	None
Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (EPA 1668C)	Soil/ Sediment	4 oz jar	Store samples in dark at $< -10^{\circ}\text{C}$ Store extracts in dark at $< -10^{\circ}\text{C}$	1 year from collection to extraction; 1 year from extraction to analysis
Polychlorinated Dibenzo-p-Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (SW-846 8290A)	Soil/ Sediment	4 oz jar	Cool to $\leq 6^{\circ}\text{C}$	None
Total Petroleum Hydrocarbons as Gasoline (Nonhalogenated Organics by Gas Chromatography, SW-846 5035/8015C)	Soil/ Sediment	3 Encore™ samples or 3 vial Terracore™ kit (2 water and 1 methanol) Do NOT add sample label to vials; vials are pre-weighed 2 oz. jar (sediment only)	Cool to $\leq 6^{\circ}\text{C}$	Encore™ or unpreserved Terracore™ samples - 48 hours from collection to extraction or freeze Preserved Terracore™ samples – 14 days from collection to analysis Sediment samples - 14 days from collection to analysis
Total Petroleum Hydrocarbons as Diesel (Nonhalogenated Organics by Gas Chromatography, SW-846 8015C)	Soil/ Sediment	4 oz jar	Cool to $\leq 6^{\circ}\text{C}$	14 days from collection to extraction; 40 days from extraction to analysis
Chemical Oxygen Demand by Semi-Automated Colorimetry (EPA Method 410.4M)	Soil/ Sediment	4 oz jar	Cool to $\leq 6^{\circ}\text{C}$	28 days from collection to analysis
Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS) (TO-15R)	Soil Gas	1 - 6-liter Summa™	Ambient	None
Volatile Organic Compounds by Gas Chromatography / Mass Spectrometry (SW-846 1311/8260D)	Leachate	2 oz jar	Cool to $\leq 6^{\circ}\text{C}$	14 days from collection to leachate; 14 days leachate to analysis
Semivolatile Organic Compounds by Gas Chromatography / Mass Spectrometry (GC/MS) (SW-846 1311/8270E)	Leachate	8 oz jar	Cool to $\leq 6^{\circ}\text{C}$	14 days from collection to leachate; 7 days from leachate to analysis

Analysis and Method	Matrix	Sample Container	Preservation	Holding Time
				extraction; 40 days from extraction to analysis
Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (SW-846 1311/6010D) or by Inductively Coupled Plasma-Mass Spectrometry (1311/6020B)	Leachate	4 oz jar	Acidify leachate to pH < 2 with HNO ₃	180 days from collection to leachate; 180 days from leachate to analysis
Mercury in Liquid Waste (Manual Cold-Vapor Technique) (SW-846 1311/7470A)	Leachate	4 oz jar	Acidify leachate to pH < 2 with HNO ₃	28 days from collection to leachate; 28 days from leachate to analysis
Organochlorine Pesticides (SW-846 1311/8081B)	Leachate	8 oz jar	Cool to ≤ 6°C	14 days from collection to leachate; 7 days from leachate to extraction; 40 days from extraction to analysis
Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzoylation Derivatization (SW-846 1311/8151A)	Leachate	8 oz jar	Cool to ≤ 6°C	14 days from collection to leachate; 7 days from leachate to extraction; 40 days from extraction to analysis
Polychlorinated Biphenyls (PCBs) by Gas Chromatography (SW-846 8082A)	Waste	4 oz jar	Cool to ≤ 6°C	None
Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (EPA 1668C)	Waste	4 oz jar	Store samples in dark; cool to ≤ -10°C Store extracts in dark at < -10°C	1 year from collection to extraction; 1 year from extraction to analysis
Polychlorinated Dibenzo-p-Dioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High Resolution Gas Chromatography/High Resolution Mass Spectrometry (SW-846 8290A)	Waste	4 oz jar	Store in dark; cool to ≤ 6°C	None
Standard Test Methods for Flash Point by Pensky-Martens Closed Cup Tester (ASTM D93) (Ignitability)	Waste	4 oz jar	Cool to ≤ 6°C	14 days from collection to analysis
Soil and Waste pH (SW-846 9045D (Corrosivity))	Waste	4 oz jar	Cool to ≤ 6°C	As soon as practicable

Analysis and Method	Matrix	Sample Container	Preservation	Holding Time
Total and Amenable Cyanide (Automated Colorimetric, with Off-Line Distillation (SW-846 9012B))	Waste	4 oz jar	Cool to $\leq 6^{\circ}\text{C}$	14 days from collection to analysis
Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides (SW-846 9030B/9034)	Waste	4 oz jar	Cool to $\leq 6^{\circ}\text{C}$	7 days

References:

ASTM – ASTM International; <https://www.astm.org/Standards>

EPA – Methods approved under Clean Water Act Section 304(h) and published in 40 CFR Part 136; <https://www.epa.gov/cwa-methods>

SM – Standard Methods for the Examination of Water and Wastewater; <https://www.standardmethods.org/>

SW-846 – Test Methods for Evaluation Solid Waste: Physical/Chemical Methods; <https://www.epa.gov/hw-sw846/sw-846-compendium>

TO – Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, EPA/625/R-96/010B; <https://www3.epa.gov/ttnamti1/files/ambient/airtox/to-15r.pdf>

Updated versions of the listed methods are acceptable if the laboratory can meet the method quantitation limit requirements in the task-specific work plans and the precision and accuracy requirements in the QAPjP.

For soils, sediments, and waste: multiple methods can be performed using the same container

Reduced sample volumes are acceptable if the laboratory can meet the method quantitation limit requirements in the task-specific work plans and the precision and accuracy requirements in the QAPjP.

Figure 2: Example Sample Label

<hr/>	
<hr/>	
Project	Job #
<hr/>	<hr/>
Date:	Time:
<hr/>	<hr/>
Sample ID:	Analysis:
<hr/>	<hr/>
Matrix:	Container:
<hr/>	<hr/>
Sampled By:	Preservative:
<hr/>	<hr/>

Figure 4: Example Custody Seal

	CUSTODY SEAL		Seal Broken By:
	Date: _____	Time: _____	Date: _____
	Name: _____		
	Company: _____		